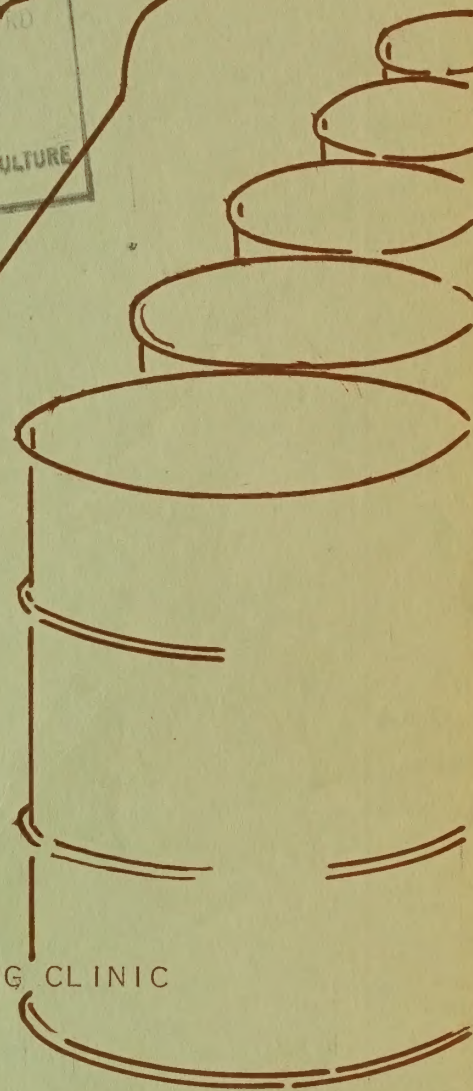
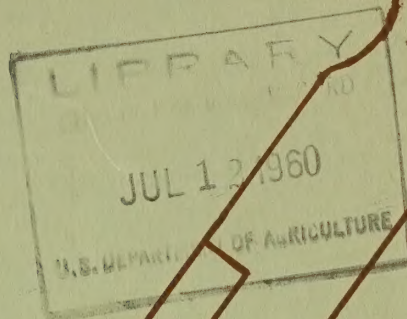


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UNITED STATES DEPARTMENT OF AGRICULTURE

AGRICULTURAL RESEARCH SERVICE

SOUTHERN UTILIZATION RESEARCH AND DEVELOPMENT DIVISION



PROCEEDINGS
OF

EIGHTH COTTONSEED PROCESSING CLINIC

AT THE

SOUTHERN REGIONAL RESEARCH LABORATORY

NEW ORLEANS, LOUISIANA

IN COOPERATION WITH

VALLEY OILSEED PROCESSORS' ASSOCIATION, INC.

FEBRUARY 16-17, 1959

FOREWORD

These proceedings are a summary of the information presented at the Eighth Cottonseed Processing Clinic held at the Southern Regional Research Laboratory, New Orleans, Louisiana, February 16-17, 1959.

Sponsored jointly by the Southern Regional Research Laboratory and the Valley Oilseed Processors' Association, this working conference was attended by seventy-four representatives of cottonseed oil mills, equipment manufacturers, users of cottonseed products, linters dealers, commercial laboratories, industry associations, and federal agencies, in addition to staff members of the Southern Laboratory. The program was arranged by staff members of the Southern Laboratory and members of the Association.

Major attention at the Clinic was focused on the sampling of cottonseed meal, the development of gossypol free cottonseed, utilization of linters, problems of cottonseed oil production, and the cleaning of cottonseed.

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OPENING REMARKS

8TH COTTONSEED PROCESSING CLINIC

By

G. E. Goheen, Acting Director
Southern Utilization Research and Development Division

Mr. Chairman and Friends: It is my pleasure to welcome you to the Southern Regional Research Laboratory on behalf of the staff of the Southern Division. We look forward to these meetings each year and to the pleasure of seeing our colleagues in the industry come back here for a visit. We are happy to have you with us and we wish to thank the Valley Oilseed Processors' Association for the opportunity that we have of working in cooperation with them in arranging these Cottonseed Processing Clinics.

Dr. Fisher, our Division Director, asked me to express his regrets that he could not be with you at this time. He is a member of a team which is in South America to obtain information on the availability of facilities and research personnel for potential Public Law 480 research projects there. During the past year the United States Department of Agriculture has been arranging for certain research projects mainly dealing with basic research to be carried out in certain foreign countries in Europe and Asia. This work is financed with funds held in these countries resulting from the sale of certain commodities exported to these countries from the United States. It is believed that this program offers an excellent opportunity to obtain the special knowledge and skill of the scientists in the foreign countries for the solution of problems facing United States agriculture. This program in no way replaces the domestic program but supplements it.

Some of you may be interested in a brief review of our organization at this location. The Southern Utilization Research and Development Division of the Agricultural Research Service comprises this building here in New Orleans - generally known as the Southern Regional Research Laboratory - and five field stations. These field stations are located in Olustee, Florida; Winter Haven, Florida; Raleigh, North Carolina; Weslaco, Texas; and Houma, Louisiana. The building in New Orleans has about 200,000 square feet of floor area and is located on about 40 acres of land at the north end of City Park. We have a staff of about 375 in this building, of which about 250 are professional people. The Southern Division is organized in five commodity research laboratories, that is, the Industrial Crops Laboratory, the Engineering and Development Laboratory, the Food Crops Laboratory, and the Cotton Chemical and Cotton Mechanical Laboratories. There are also two Pioneering Research groups which emphasize basic research. One of these is the Seed Protein Group and the other is the Plant Fibers Group. Development work is emphasized by the formation of task groups and teams through cooperation of personnel in the ED Laboratory with the appropriate commodity research laboratory.

While about 75% of the work of the Southern Division is concerned with cotton lint and cottonseed, the remaining work involves many other commodities grown in the South. For example, tung, sesame, peanuts, jojoba, citrus fruits and Southern vegetables such as sweetpotatoes and pickles, rice, pine gum, sugarcane and certain new or replacement crops. Castor research which was carried out in our Division during the last few years was transferred to the Western Division in California as of the first of this year.

In a review of accomplishments for last year the Southern Division listed 124 items - either new developments or significant progress in developments previously reported. New information developed by the Division was published in 137 papers during 1958. Sixteen patents on previous developments were granted during the year and 21 new patent applications were filed.

Many of the achievements relating to cottonseed processing and the resulting oil and meal will be discussed in the excellent program which has been worked out for the current meeting. However, I would like to mention briefly some of the achievements of the Southern Division in other areas.

New developments in the field of wash-wear cotton fabrics have been a feature of our cotton research. Wash-wear cottons though not perfect in their present state of development, have met with enthusiastic consumer acceptance. We are aiming now for the perfect wash-wear garment -- a garment that can go to the laundry, or into the washing machine and come out looking smooth and crisp without even a touch-up from the iron; a garment with flat, unpuckered seams, and sharp, long-lasting creases where creases are wanted. Such an achievement should open some important new markets for cotton, especially in cotton dress trousers for summer wear.

The Defense Department is interested in some of our other developments, especially fabric treated for flame resistance, resistance to oil, and resistance to penetration by acids.

Improving resistance of cotton fabric and thread to degradation from weathering, mildew, and heat has come in for attention, and ways of prolonging the life and beauty of cotton awnings have been studied. Evaluation tests here have shown that by proper selection of coating pigments, awnings can be protected from solar degradation, and their appearance preserved, for as much as three years.

Mechanical processing of cotton has not been neglected, and several advances in this field have been reported. The SRRL Opener-Cleaner, announced in October 1957 is in operation in a number of textile mills. Some of them report savings up to \$100 per day per machine, and fabric quality increases up to one full grade.

The newest development is the flatless or granular card. The cotton textile industry reports this machine gives 4% less fiber waste, 10% fewer neps, and produces a more uniform sliver. Commercial manufacture of the apparatus by a number of textile machinery firms is already assured.

A new bale-breaker-blender will open and mix cotton from as many as 49 bales into a homogeneous blend. It has been demonstrated that fine and coarse cottons can be blended to produce satisfactory yarns. This promises to furnish an outlet for qualities of cotton now hard to market.

We believe that Government research pays for itself many times in savings to the taxpayer. For example, a recent survey of new or improved products which have been commercialized as a result of the work of our scientists showed that they now have actual annual sales of over 265 million dollars. Just the taxes returned to the Federal Government on these new products more than pay for all of the utilization research which has been carried out by the Southern Division.

As program administrators and scientists we are constantly striving for the most efficient way of solving your problems through research. We appreciate your advice and suggestions. We give you a cordial invitation to visit our laboratories any time, either individually or in groups.

In closing I would like to express thanks to the Valley Oilseed Processors' Association and to its officers. I would also like to thank the members of our staff who helped arrange and plan this clinic, particularly Mr. Persell, Mr. Gastrock, Mr. Patton, Dr. Pollard, and Mr. Knoepfler.

RESPONSE

By

Robert F. Patterson, Chairman
Program Committee
Valley Oilseed Processors' Association, Inc.

Thank you, Dr. Goheen, for such a warm and sincere welcome.

The presence of so many here this morning - in the face of some rather difficult adjustments in our industry - testifies to the interest in these meetings.

It is therefore my happy privilege - on behalf of the Valley Oilseed Processors' Association and these assembled here to thank you and your staff for the arrangements for our meeting and for your continued interest in the problems of our industry.

SAMPLING BULK-LOADED MEALS

By

M. H. Fowler
Buckeye Cotton Oil Company

If all the cottonseed meal in a 50 ton car were divided up into samples for nitrogen analyses, there would be enough portions for 26,635,000 individual determinations. When all the reports came in from the several hundred laboratories you would have to use, they could be averaged and, there is no doubt, the average would be precise. The lowest result would be about 5% protein and the highest, 60% protein. Now, if we make some reasonable assumptions, we can estimate that the analyses will be distributed about as shown on the bar graph (Figure 1). If the meal averaged 41% protein, half the results will fall between 36.5 and 45.5% protein. The range 27.9 to 54.1% will include 95% of the total; and so on.

This is the situation you would face if the chemist simply weighed his sample for analysis directly from the loaded car. It's an absurdity, of course, but it serves to show the nature of the commodity with which we are dealing.

The 5% protein material is almost pure hulls and the 60% protein material is pure extracted kernel. Only random chance mixing is assumed.

When oilseed meals were first loaded and shipped in bulk, it was the practice to convey the meal to the center of a car and shovel it by hand to the ends. In this way, some mixing occurred. Equally as important, very little "unmixing" took place. The load was finished off approximately level from end to end and was reasonably homogeneous. Under such conditions sampling presented only minor difficulties. For years the industry got on well with a sampling method adapted directly from the standard grain sampling methods long in use.

Then two things happened that added difficulty to the sampling problem and introduced frustration into the business of buying and selling meals.

1. Solvent extraction process began to remove more of the oil from meals increasing the tendency of the meal and hull particles to segregate when handled.
2. Bulk-loading methods were introduced that actually separated dense and light particles in the process of loading. When a blower or sling loader is used to **load** cars, the largest and densest particles are projected farthest. The less dense hull settles nearer the loader. You have all seen this I'm sure and you are familiar with the experimental use of this principle to clean cottonseed and to separate them by density. Such loading methods not only leave layers

TABLE 1

Probe No.	Car 1	Car 2	Car 3
1	44.25	39.06	45.81
2	45.69*	45.19	47.44*
3	40.31	41.69	41.88
4	41.25	46.19*	45.19
5	44.00	42.88	38.44
6	40.75	41.38	42.44
7	40.25	41.88	46.44
8	44.25	40.25	40.25
9	39.69*	41.19	38.38
10	39.75	40.44	41.75
11	40.25	40.75	41.00
12	40.56	41.94	39.06
13	40.50	40.63	40.88
14	40.50	38.81*	41.81
15	40.88	39.06	41.31
16	40.00	38.81	41.25
17	40.13	40.56	37.75
18	40.00	40.13	37.19
19	40.94	40.63	38.88
20	40.75	40.75	38.50
21	42.06	41.69	37.94
22	40.44	40.81	36.88*
23	41.44	42.50	38.88
24	41.13	41.69	39.50
25	39.94	39.94	38.88
AVERAGE	41.19	41.15	40.72
COMPOSITE	41.44	41.00	39.81
DEST. SAMPLE	41.74	39.22	40.79
AUTO. SAMPLE	40.56	39.44	40.94

and pockets of separated hulls and dust, but they leave the profile of the loaded car unfavorable to good sampling. The ends of the car are filled nearly to the roof and the center varies from a few inches to about two feet in depth.

It soon became obvious to the N.C.P.A. that the five probes of the Official Method simply weren't a reliable sample.

In 1955, the Association changed their rules to require 25 probes. As might be expected, this method, when followed, gives a noticeable improvement in the agreement of two samples taken from the same car. Original and destination samples, however, still varied widely in many cases.

There is a committee in the American Oil Chemists' Society studying this problem of sampling. The A.O.C.S. has already approved the use of an automatic sampling device which they recommended. This committee has collected some enlightening data on the variance in the probe sampling method, which I would like to discuss with you.

Table 1 shows how individual probes within a single car sample vary in protein content. The average range of individual probe samples in the three cases shown was 8% protein.

The standard deviation, combining the three sets of data, is $\pm 2.06\%$ protein. From this we can infer something about the reliability of the sample average. For example, we can say with about 95% certainty that the average of 5 probes will lie within $\pm 1.84\%$ protein of the true average of the car (Table 2). The average of 25 probes will lie within $\pm .82\%$ of the true average. This is not a comforting thing to say about a sampling procedure, but it's the best that can be said for the probe sample as we take it now. From this it is readily apparent that we cannot take less than the prescribed 25 probes and probably should take more.

TABLE 2

Reliability of Average - Probe Sampling				Probability	
				<u>50%</u>	<u>95.5%</u>
Avg. % Protein -	5	probes		$\pm .62$	± 1.84
" " "	- 10	probes		$\pm .44$	± 1.30
" " "	- 25	probes		$\pm .28$	$\pm .82$

We have mentioned the automatic sampler only briefly. One may well ask how it compares with the probe method. The files of the A.O.C.S. Committee

on Bulk Meal Sampling contain some comparative data on automatically taken samples versus various schemes of probe sampling. There is no way to tell from these data which method is more nearly correct. However, it is possible to see that the agreement is not always satisfactory. It may be possible eventually to devise a test which will actually compare the accuracy of the automatic sampler and the various probe-sample methods. For the present, however, arguments in favor of the automatic sampler are based on logical considerations:

1. At no time after meal leaves a "slinger" does it exist as a homogeneous mass in a bulk-loaded car.
2. Sample probes cannot reach some parts of bulk-loaded cars.
3. Because of the uneven profile of bulk-loaded cars, it is very difficult to assure that proportional weights of samples are taken from each part of the car.
4. If the loading rate is uniform, the automatic sampler described in A.O.C.S. Method Ba 1-38 cannot fail to take a gross sample representative of the whole car.

Taking the gross sample is not all there is to the job. Reducing this sample to laboratory size presents problems. The chemist is only going to weigh 1.7032 grams (about .06 oz.). Obviously, the steps in sample reduction must be precise and be carefully taken.

We wanted to know what variance to expect under carefully controlled conditions using two methods of sample reduction. First, we took a single sack of meal prepared in a production batch mixer and divided it into 32 equal parts with a riffle. Triplicate protein determinations were made on each portion.

Next some meal of the same type was finely ground (100% - 20 mesh) and mixed in a MacLellan mixer. Sixty portions were grabbed from the mixer. Single analyses were made on each grab.

In order to get an idea of the within-laboratory variance of the Analytical Method, 90 single analyses were made on 6 very finely-ground and well-mixed, small meal samples. The standard deviation thus derived should be free of most of the sampling variance and is used here as an estimate of the standard deviation of the Analytical Method itself.

From these three experiments, we derived a table which tells quite a bit about the precision of protein determinations. Part of this table is reproduced on Table 3. This chart shows that with proper care in reducing samples very acceptable precision can be attained. For example, if the gross sample truly represents the whole, we can be 95.5% certain that a single analysis will be within $\pm 0.57\%$ of the average if we grind the sample and mix it in a MacLellan. The result will be within $\pm 0.72\%$ protein of the average if we reduce the gross sample by riffing without grinding.

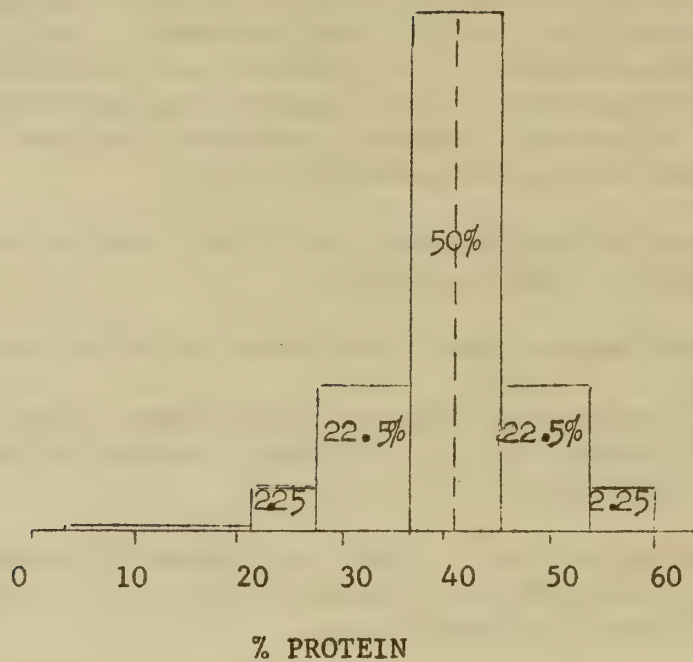


Fig. 1. Distribution of results from protein analyses.

TABLE 3

Probable Deviation From Average, \pm % Protein

No. of Determinations In Average	50% Probability				
	1	2	4	10	25
Small Sample Fine Grind	.13	.08	.07	.04	.03
Gross Sample Ground & Mixed	.19	.13	.10	.06	.04
Gross Sample Riffled	.24	.18	.12	.08	.05

No. of Determinations In Average	95.5% Probability				
	1	2	4	10	25
Small Sample Fine Grind	.39	.28	.20	.12	.08
Gross Sample Ground & Mixed	.57	.40	.29	.18	.11
Gross Sample Riffled	.72	.51	.36	.23	.14

Bulk Car Samples - From Table 3
 Avg. 5 probes - \pm 1.84 % Protein
 Avg. 10 probes - \pm 1.30 " "
 Avg. 25 probes - \pm .82 " "

For comparison, the variance from the probe sampling experiment described previously is shown at the bottom of Table 3. You will note that the standard 25 probe sample may vary from the average by $\pm .82\%$ protein.

We have said that the standard deviation of the single probe analysis from the average for the car is $\pm 2.06\%$ protein and the standard deviation for the Analytical Method is $\pm 0.20\%$ protein. Then the standard deviation of the sampling and sample preparation is $\pm 2.05\%$. Therefore, our most significant improvements must lie in sampling. The simplest approach to this kind of problem is to increase the number of increments taken as gross sample. The automatic sampler can easily be set to take 100 cuts (as opposed to the 25 probes). If the variance were not improved at all - and we think it would be - the standard deviation would be reduced from $\frac{2.05}{\sqrt{25}} = \pm 0.41$ to $\frac{2.05}{\sqrt{100}} = \pm 0.21$. We would then have a 95.5% probability of getting our answer within ± 0.42 of the average.

These facts taken all together mean that we can expect some significant improvements in protein analyses of bulk shipments by attention to a few details:

1. Bulk-loaded meal is non-homogeneous. The gross sample must be very carefully taken. The A.O.C.S. approved Automatic Sampler Method is the most reliable means we know for taking the gross sample.
2. Fine grinding of the entire gross sample would improve accuracy appreciably.
3. Mixing in an efficient mixer and grabbing out the laboratory sample is preferred to riffing or quartering especially if the gross sample is finely ground.
4. If the car sample must be taken by probe, the surface of the meal should first be leveled. No less than 25 probes should be taken.

By analogy, all the points mentioned here apply to soybean meal also, especially to the 44% protein product. A very large portion of 44% soybean meal now contains mill feed or ground hulls taken out of the stream when high protein meal is produced. These hulls tend to segregate upon loading even worse than cottonseed hulls so the problems in sampling the two products are the same.

RESIDUAL SOLVENT IN SOLVENT EXTRACTED MEALS

A Review By

E. A. Gastrock and J. J. Spadaro
Southern Utilization Research and Development Division

My purpose this morning is to not only review briefly some factors pertaining to residual solvent in meal, but also to stimulate your thinking so that, during the panel discussion to follow, some suggestions or comments may be made that will contribute to solving the problem of determining residual solvent in meal.

Residual solvent in meal, in quantities sufficient to produce explosive mixtures with air, has been recognized as an extremely important problem from the beginning of solvent extraction operations for cottonseed and soybeans as well as other oil-bearing materials.

Thousands and thousands of tons of solvent extracted meal have been produced annually. Although there have been relatively few explosions and fires, where they have occurred, serious loss of life and property damage resulted. For this reason it is the aim of the industry to eliminate entirely the hazard of residual solvent in meal by some simple and reliable method of detection of solvent in meal.

In order to eliminate any possibility of explosion, solvent in extracted meals, must be removed, not to the extent of 99 44/100%, but to the extent of 99.9939% which means essentially 100%. Some simple calculations will show you why.

Let's consider a shipment of 80,000 lbs. of meal in a boxcar.

Volume of a boxcar is 2600 cu. ft.

Volume of 80,000 lbs. meal is 914 cu. ft.

Remaining air space in car is 1686 cu. ft.

The Lower Explosive Limit (LEL) of hexane is 1.2% of solvent vapors in air by volume.

Therefore, $\frac{1686 \times 1.2}{100} = 20.2$ cu. ft. of hexane vapors required to

reach the lower explosive limit in mixture with the air in the boxcar.

Mol. Wt. of hexane is 86

1 cu. ft. = 28.3 liters

lbs. of hexane is 20.2 cu. ft. vapors =

$$\frac{86}{22.4} \times \frac{28.3 \times 20.2}{453.4} = 4.88 \text{ lbs.}$$

Percentage-wise, the 4.88 lbs. of hexane in 80,000 lbs. of meal is only $\frac{4.88 \times 100}{80,000} = 0.0061\%$. Or, as I mentioned before, 99.9939% of

the solvent in meal must be removed to stay below the L.E.L.

The Upper Explosive Limit (U.E.L.) is 6.9% solvent vapors in air by volume. Similar calculations show that only 27.9 lbs. of solvent in 80,000 lbs. of meal or 0.0349% residual solvent is required to attain the Upper Explosive Limit (U.E.L.).

Some of you may say that perhaps, it is best to leave 0.1 or even 1.0% solvent in the meal. Then it will be above the explosive limit. That is true if all the vapor was put in the air at one time but, as solvent starts to evaporate, the ratio of solvent vapors to air will pass through the explosive limits. Then you are likely to have both an explosion plus a more quickly spread fire--the excess solvent that didn't take part in the explosion will burn.

Also, when opening the door of a boxcar containing solvent vapors above the U.E.L., a mixture of these vapors with the outside air, can form the proper explosive ratio, and at a very critical time.

The methods currently used by the industry for determining residual solvents in meal can best be reviewed by summarizing the findings the AOCS Subcommittee on Residual Solvents in Meals which is headed by Mr. E. A. Gastrock as chairman. A member of that committee, Mr. Ross Brian of Central Soya Company, sent a questionnaire to 83 solvent extractor operators. Forty-three replies were received, of which 23 indicated they used a method for determining residual solvent, and 20 stated they did not use a method. Of the 23 only 3 use a quantitative method, the other twenty use qualitative methods. Eight indicated that the method they used was satisfactory.

The general concensus of industry is, however, that a more satisfactory method is required.

The two most used qualitative methods of determining residual solvent according to the reports were: (1) a sample of the extracted meal is placed in a tightly covered metal can. Sizes of cans differ. Various means are used for mixing the contents and various times are allowed for equilibration. In some cases equilibration takes place in a warm atmosphere. The lid is partially lifted and a small flame is inserted into the top of the can. If a pop or flash results, it is assumed the residual solvent is too high.

One company had attempted to establish a semiquantitative test using the metal can-pop method. Type of flame obtained was to indicate the approximate amount of solvent present.

(2) In the second method a combustible gas detector such as the MSA (Mine Safety Appliance) equipment is used for sampling the atmosphere within a conveyor continuously or for sampling the atmosphere within a jar containing a sample. Several arrangements of the MSA in conveyors are used. The alarm is generally set to sound when the concentration reaches 40% of the Lower Explosive Limit. The main difficulty of this method appears to be clogging of the intake due to dusty and humid conditions of the atmosphere within the conveyor.

A quantitative method used by one company only occasionally, involves two tests - one for the determination of moisture by the toluene distillation method and the second for the determination of moisture and volatile by the oven method. The difference is recorded as the hexane content of the meal. This method is time consuming, and the accuracy obtained has not been fully determined.

One organization reported a method in which nitrogen is passed through a pint sample at 150° F.; then dried in two columns and absorbed on activated carbon. The activated carbon is then weighed to determine the amount of hexane absorbed. Another company uses the azeotropic distillation method.

A method using gas chromatography has been investigated by at least one person. This method does not appear too attractive for mill use due to the cost of equipment and the need for a specially trained operator.

A practical method for use in solvent extraction plants has reportedly been developed by a Dr. Paul Van Der Voort, of Anvers, Belgium. But to our knowledge he has not published the method.

There are several methods, based primarily on analytical procedures, that could possibly be investigated as methods of determining both quantitatively and qualitatively residual solvent in meals. Some examples are:

- (1) Electrical detectors of which there are three groups: combustion, catalytic, and diffusion type. Many of these methods have been developed for methane and perhaps they could be modified for use with hexane. (MSA and Wheatstone bridge types come under this category.)
- (2) Adsorption methods which include absorption media such as silica gel and activated charcoal.
- (3) In the third group are the physical chemical methods of which there are several:
 - (a) Gas Interferometer - which compares refractive indexes of two specimens of gas or vapor one of which is a gas of known refractivity.
 - (b) Conductivity methods.
 - (c) Adsorption of radiant energy.
 - (d) Vapor pressure methods in which a freezing agent is used to condense vapors.

A semiquantitative method that has been considered by the Engineering and Development Laboratory here is the use of an apparatus analogous

to the closed cup tester for oil which has proven satisfactory for checking the oil prior to shipment. The apparatus consists of a metal container with a flange top which contains a rupture disc, a spark plug, a thermometer, and a vent. Heat from a hotplate is applied so that the temperature of the meal rises slowly. An electric spark from a distributor and coil is applied at frequent intervals.

With such an apparatus, a correlation might be worked out between the amount of sample required to obtain an explosion, and the approximate solvent content in the meal. For example:

A 300 gram sample in a 1000 cc. container should give an explosive mixture when the meals contain between 0.01 and 0.07% hexane. Smaller or larger samples would be used for higher or lower hexane percentages.

Work with this apparatus has been of exploratory nature only. Although we have been able to obtain some explosions, proper correlation has not been obtained. We have, however, learned several things: (1) a more efficient transfer of heat to the sample is required; (2) container should be airtight; (3) a mixer should be included in the apparatus; and (4) spark should be dependable and located in the proper spot.

I'm going to take a few minutes to demonstrate the apparatus to you, but first, I would like to leave with you a statement of the objective of the AOCS Subcommittee on Residual Solvent in meal - which is "to develop and/or select a suitable method for the detection and measurement of residual solvent in solvent-extracted meal so that producers, shippers, handlers, and users of solvent extracted meal may have confidence in the safety of that commodity". In other words, the industry still needs a rapid and reliable method for determining residual solvent.

MILL SCALE MATERIAL BALANCE

By

J. H. Brawner
Wesson Oil & Snowdrift Company

The subject that was assigned to me was "Mill Scale Material Balance." However, we rarely have serious trouble in reconciling total pounds sold with total pounds purchased.

What we ordinarily mean when we talk about "material balance" is the relationships between the actual oil and meal yields and what the official methods of analysis indicate.

Unless we obviously slip-up somewhere, we usually are fairly successful in balancing protein sold with protein purchased in seed.

It is failure to reconcile the oil produced and oil left in products with oil in seed purchased that most often seems to worry processors. In the first place, oil is our most valuable product, and a few pounds per ton can represent a considerable amount of money. In the second place, when we make an oil balance we usually consider only the oil produced and the percents oil in hulls and meal. We disregard the oil in lint and oil discarded in trash. Therefore, most of the time we have a so-called unaccounted-for loss of roughly five pounds, or perhaps a little less, and when this original loss is enlarged through some error, it begins to look pretty big to us.

Ocassionally, and without apparent reason, we have some serious unaccounted-for losses. Since the yields of products plus discarded trash and moisture loss usually check the tonnage purchased reasonably well, and since our protein balance usually is pretty good; oil discrepancies under these circumstances suggest that we must have some "error" in percent oil in seed, percent oil in meal or percent oil in hulls, or possibly all of these.

I suppose that most of us sample the seed crushed each day. In our cases where we have had large unaccounted-for losses, many times we have found that the average percent oil in seed crushed for the season checks quite well with the average of official grading analyses. In fact, I do not remember more than one or two cases where there was much difference between these two averages.

If we accept the full implication of this statement, then it seems that any "error" in oil in seed must be a persistent error caused by failure of the analytical procedure to measure the same thing that the mill actually obtains as "oil". This also can be said of the analysis for "oil" in meal and oil in hulls.

As far as hulls are concerned, it seems to me that most of the chances of trouble lie in inaccurate sampling rather than in some queer situation where the analysis does not measure all the "oil" that is present. Most oil mill people know this well, so it is hard for me to believe that we may frequently have as much as 10 to 20 pounds of unaccounted-for "oil" due to poor hull sampling. However, it will not hurt to emphasize the fact that accurate hull sampling is extremely difficult and everyone should take considerable pains to get good samples.

Let us see if there is any evidence that "oil" in seed, "oil" in meal, and "oil" yield may represent different things at times.

In the first place, most of us know that solvent extraction checks with the seed and meal analyses very well. In fact, solvent extraction with hexane seems to remove a little more material than the petrolic ether used by the control laboratories.

On the other hand, Expeller and Screw-press units seem to take out less than the laboratories indicate is removed. Hydraulic units are somewhere between solvent extraction units and the mechanical presses, and often the hydraulic oil yields check with the laboratory results quite well.

About two years ago, we reported that our operations at that time indicated that mechanical presses had an average unaccounted-for "oil" loss approximately 2.3 pounds greater than hydraulic mills.

As a further check, we have reviewed our results for the past two years. We find that for 35 mechanical press cleanups and 22 hydraulic cleanups during the past two years, the average difference is 2.1 pounds, which checks the previous figure quite well.

In passing, it is interesting to note that during the two years, the mechanical press unaccounted-for losses varied over a much wider range than hydraulic unaccounted-for losses.

It seems to me that in order to get at the causes of variations in unaccounted-for "oil" losses, we should start out by trying to learn how the "oil" extracted from seed during analysis and "oil" extracted from meal during analysis differ from each other and from the corresponding "oil" actually removed in the mill. We should also try to find out if fatty material is made insoluble in petrolic ether during processing.

Since the widest variations in unaccounted-for losses occur in mechanical pressing, this particular process might yield information fastest.

In closing, I would like to mention cleanup tests. A cleanup test is a test whereby you carefully weigh and sample a definite tonnage of seed, and process it separate from any other seed; carefully separating, weighing and sampling the products. At the completion of the test, an attempt is made to obtain an "oil" balance, a protein balance, and so on. Such tests are very difficult and expensive to make and require supervision far beyond that available at most oil mills. Furthermore, most mills do not have the seed and products storage and handling facilities to give the needed accuracy for a short test. For these reasons, I feel discouraged about the value of cleanup tests, and I believe that the study of the analytical extraction processes is the far more fruitful route to the solution of our unaccounted-for "oil" loss problems.

PANEL DISCUSSION - MORNING SESSION - FEBRUARY 16, 1959

PANEL - Verdery, M. C., Moderator
Brawner, J. H.
Fowler, M. H.
Mays, J. R., Jr.
Smith, Allen
Spadaro, J. J.

Verdery: We will open the discussion with Mr. Fowler's paper. We have had plenty of trouble at our mills with this problem. We have not only had trouble with cars, but with tanks and bins. On one occasion, we filled a 200-ton tank with meal. We took samples from every 6-inch stratum. The protein in these samples varied from 37-46%. Samples taken from the product stream varied 2%.

Mr. Verdery quoted from Table "A" (opposite page) showing actual average protein for the season for three typical oil mills along with the percentages of production meal running above and below the desired 41% protein along with estimated dollar losses incurred in an effort to maintain 41% protein.

It will be noted from Table "A" that Mill "B", which averaged approximately 41% protein for the season, actually shipped 42% of their meal below 41% and 21% over 41.50%

Mill "C", making a special effort not to make any shipments under 40.50%, actually shipped only 8% of their production below this level but in so doing shipped 65% in excess of 41.50%. It will be noted from the tabulations that the expense of maintaining the protein on the high side is relatively costly and clearly illustrates the necessity of more accurate protein control.

Verdery: Mr. Mays, how do you feel about this sampling problem?

Mays: I think that many mills are not taking 25 probes per car for various reasons. Sometimes they can't be taken, by reason of the way the car is loaded. Meal samples may actually be taken from only one or two points in the car. This is one of the main causes of trouble. The use of an automatic sampler by the producer would eliminate this trouble. However, if 25 probes of a car were actually taken, we would have much better agreement.

McClure: If the producer settles on an automatic sampler, how can he convince the buyer he should use an automatic sampler?

TABLE "A"

PROTEIN CONTROL
(Season 1957-58.)

	<u>MILL "A"</u>	<u>MILL "B"</u>	<u>MILL "C"</u>
1. Desired Protein	41.00%	41.00%	41.00%
2. Production Protein - Avg.	41.66%	41.04%	41.99%
3. Value of Excess Protein	\$9,200.	\$562.	\$46,600.
4. Tons Crushed	21,700	20,800	66,700
5. Cost - \$/Ton	\$0.424	\$0.027	\$0.700
6. Highest Protein	45.66%	42.87%	44.23%
7. Lowest Protein	39.81%	39.41%	39.75%
8. Annual Range	5.85%	3.46%	4.48%
9. Production below 41%	15%	42%	19%
10. Production above 41.50%	73%	21%	65%
11. Production below 40.50%	12%	29%	8%
12. Cost to Guarantee all Protein above 41%	\$16,500.	\$22,100.	\$58,800.
\$/Ton	\$0.760	\$1.062	\$0.883

Fowler: I see no reason why the buyer couldn't use an automatic sampler when he unloads the car. Most buyers, however, want a sample before they unload. Oil is treated that way. It appears likely that cars will have to be sampled by probe, if they are to be sampled before unloading.

Brawner: The problem of particle size segregation aggravates the sampling problem. Solvent extracted meal is the most difficult since it segregates badly. Characteristically it is dry, dusty, **nonuniform**, and its hull particles tend to rise up to the top. Some work is needed on ways of preventing this segregation in solvent meal. Some methods of loading make segregation worse, particularly the kicker-type. We discarded kickers in preference to portable conveyors. The conveyors were operated as close to the floor as possible.

Gastrock: I wish to make an observation that the present tendency toward producing a higher protein content meal should also tend to reduce the separation of hull particles from meal particles. In other words, if less hulls are present there will be less separation.

Woodruff: Mr. Mays, how close are we to the use of automatic samplers?

Mays: It is in the official methods book right now if the producer wants to use it. I suppose its use would depend on an agreement between the producer and the buyer. An automatic sampling method was put in at the same time the rules were changed to require 25 probes. As with all the other rules, the one used depends on agreement between buyer and seller.

Wamble: I would like to know where the licensed sampler comes in if automatic samplers are used?

Mays: This is speculation, but it seems to me that the automatic sampler should discharge into a NCPA sealed container. The licensed sampler would then break the seal and prepare the sample for shipment.

Wamble: It is necessary to maintain a check on automatic samplers. Mechanical devices are not infallible, you know.

Smith: I would like to know what methods various mills are using to get uniformity in bagged meal. If the bagged meal is not uniform, the state inspector may get a sample of low protein meal at some country store, and then we have to pay a penalty. Whereas, if the whole car were sampled it would be O.K. At one of our mills we have taken some steps to improve uniformity. What have you done, Mr. Wiley, to get better uniformity?

Wiley: I think you are better qualified, Mr. Smith, to discuss that matter. You planned it and looked over my shoulder while I made all the changes.

Smith: Mr. Brawner, you people have done the best job I know of--far better than ours. How does your meal loading range run?

Brawner: Satisfactory. We grind as produced, and do not store in bins. This prevents segregation in the bins. Lint beater bran is blended with the meal after grinding. Then we do the final protein adjustment with the uniform method hull bran. We make our own hull bran. The hull bran is added to the meal stream by using variable speed drive feeders. It requires good equipment and close supervision.

Verdery: What does your protein average out? Do you shoot for 41% or slightly higher?

Brawner: It is necessary to keep on the high side.

Verdery: One of our mills shot for the high side and averaged 41.66%. Fifteen percent of the meal was over 41%, 73% was above 41.5%, and 12% was below 40.5%. At another mill, we averaged 41.04% for the season. Forty-two percent of the meal was slightly over 41%, 29% was below 40.5%. About 30% got cheated, 50% was in the middle, and 20% was over. The problem is to level the thing out.

Smith: Our average comes out right on 41% over a month's period. The trouble comes in in variation of the individual cars. This leads to a two-way loss--we don't get paid for the over protein, but we do get penalized for the under. We hope, by changes made recently, to get 50% of our outgoing cars exactly on 41%.

Verdery: The proper protein control could make the mills as much as 50¢/ton on the meal.

If there are no more questions, we will move on to Mr. Spadaro's paper. Does anyone have any questions about residual solvent in meal?

Brawner: I agree that solvent in meal is a most important problem. We have had some very difficult experiences. Initial explosion of a small amount of solvent puts cottonseed dust in the air. Cottonseed meal as dust is as explosive as dynamite and actually worse than coal dust. Meal handling is the big problem. We use ~~explosion-proof~~ equipment. We find that our desolventizing equipment forms balls which are high in solvent, and which our solvent detection equipment cannot find. Mr. Spadaro, will your test apparatus detect the solvent in these balls?

Spadaro: The inclusion of a mixer to stir the sample in the cup could breakup these balls and make this solvent detectable.

Smith: Can a fellow with an average sense of smell detect the presence of solvent in the danger zone?

Spadaro: Yes, if it's above the upper explosive limits. No, if it's below the lower explosive limits.

Fowler: The nose becomes desensitized after the first sniff, then you have to wait awhile. We have MSA elements along the conveyor, but the balls are a problem. Solvent in the storage bins and conveyors is more of a problem than solvent in the loaded cars.

Gastrock: The problem of solvent in the cars was used only as an illustration. We recognize that a solvent problem exists throughout the meal handling portion of the plant.

Spadaro: I would like to point out that a sample directly from the dryer may have no solvent odor, and that it may not be detected in the conveyor by the MSA equipment. The reason for this is that the solvent in the interior of meal particles and balls requires time to diffuse to the surface.

Verdery: I gathered from your talk that you do not consider the MSA detectors satisfactory. Is it the best equipment available at the present time?

Spadaro: My report is based on a survey, which included users of MSA equipment. Some of these are satisfied with it, some are not. The most common trouble is that the nozzle clogs up with dust in the conveyors.

Moore: MSA shows free vapor, but does not measure that in meal particles and balls. I recently saw a device used universally in Europe. It consisted of sealed glass tubes half filled with brown crystals and half filled with white crystals. You break the tip off the tube, and attach a hand aspirator to it. Air is drawn into the tube by squeezing the bulb, counting the number of squeezes, until the white crystals become the same color as the brown. By referring to a chart provided with the instrument, you can determine by the number of squeezes the amount of solvent in the air. It sells for about \$40 and is claimed to be accurate.

Gastrock: I know of one company, which uses the MSA detector to check vapor in alternate bins at one hour intervals. The first bin is filled and permitted to stand for one hour while the second is being loaded. This gives time for the solvent to diffuse out of the meal and helps to solve the diffusion problem.

Verdery: If there are no further questions on solvent in meal, we will go on to Mr. Brawner's paper of mill scale material balances. Mr. Brawner, do you have any comments to make?

Brawner: I believe I said in my talk that we had found a correlation between oil in the seed bought and oil loss in the mill. We took 33

full scale cleanup tests and based a statistical analysis on this data. The only positive correlation we found was between percent oil in seed crushed and unaccounted-for oil loss.

When a mill completes its annual crush, it becomes possible to calculate the actual yields of linters, hulls, meal, and oil for the crush.

By subtracting the pounds of oil produced per ton of purchased seed from the total pounds of oil in seed (as determined from the official seed analysis), and "Oil Difference" is obtained. This supposedly represents true processing efficiency.

Practically all mills have meal and hulls analyzed for percent oil. By multiplying the season's actual yields by the corresponding percentages of oil for the season, the pounds of oil in meal per ton of seed, and a corresponding pounds of oil in hulls are obtained. When these are subtracted from "Oil Difference", a so-called unaccounted-for oil "loss" is obtained.

For 33 Expeller and Screw Press mill-seasons, simple correlation coefficients were obtained for unaccounted-for oil loss and FFA in oil in seed crushed, percent oil in seed crushed, moisture in seed bought, and Oil Difference. The simple correlation coefficients were as follows:

Unaccounted-for oil loss Vs. moisture in seed bought	0.1822
Unaccounted-for oil loss Vs. FFA in seed crushed	0.2856
Unaccounted-for oil loss Vs. percent oil in seed crushed	0.4034
Unaccounted-for oil loss Vs. Oil Difference	0.6559

According to Fisher's tables of significance, only oil in seed and oil difference had any significant relationship to unaccounted-for loss.

It was felt that since oil difference included unaccounted-for loss, its correlation with unaccounted-for loss was questionable.

A new set of correlations was made between unaccounted-for loss, percent oil in seed bought, and pounds of oil in meal. Meanwhile, another season's data became available, so there were 46 mill-seasons used this time. The simple correlation coefficients were as follows:

Unaccounted-for oil loss Vs. percent oil in seed bought	0.3607
Unaccounted-for oil loss Vs. pounds of oil in meal	0.0542

According to Fisher's tables, only the first correlation coefficient is significant.

From these data, it may be concluded that of the factors studied, percent oil in seed probably has a definite relationship to unaccounted-for oil loss.

Smith: Did I understand you to say that the only positive correlation you found was between oil in the seed bought and oil loss?

Brawner: Yes, that is what our data shows.

Smith: You did not find any correlation between cooking temperature and oil loss?

Brawner: Our results indicate that the only significant correlation existed between the percent oil in seed purchased and the unaccounted-for oil loss.

Verdery: Since there are no more questions, I will now turn the meeting back to Mr. Woodruff.

Woodruff: Even in as late a period as our grandfathers, a man died in about the same economic and technological environment as that in which he was born. Throughout the years of his life he used similar tools, lived in the same kind of house, ate the same food and received about the same wages for his work with little or no advancement in his manner of living or his position in life. The dramatic effects of change were not a problem to him.

Contrast that life with the present in this country, when such tremendous technological changes are taking place. Instead of living in a static environment we only know that what is familiar today will be discarded for something better tomorrow. We are living in a dynamic age and the only thing that we are quite sure of is that what we have already accomplished will be dwarfed by developments yet to come.

It has been estimated that twenty years from now there will be sixty million more people in this country than there are today, but that there will be only slightly more workers between the ages of twenty and fifty. Since the average worker today produces enough to provide for himself plus 1 1/2 other persons--then twenty years from now the average worker must produce enough for himself and 3 1/2 other people. In other words, it appears that possibly, within twenty years we must find ways to double productive capacity or our standard of living will decrease.

We must all understand that we in our business, or any other business, have no option as to whether or not we choose to accept progressive ideas and adopt them. If we are to remain in existence, then progressive adoption of new ideas and methods and sciences is mandatory.

CURRENT UTILIZATION OF COTTONSEED OIL AND MEAL

By

K. M. Decossas, E. F. Pollard, and E. L. Patton
Southern Utilization Research and Development Division

Cottonseed Oil

More than half of the factory consumption of cottonseed oil is being winterized for use in cooking oils, salad oils, mayonnaise, and related products, and the use of cottonseed oil in this market has been increasing.

The other important uses of cottonseed oil are in shortening, margarine, and mellorine. Vegetable oil consumption in shortening is decreasing, being replaced by animal fats and oils. Because of the interchangeability of most fats and oils in this end use, the relative prices of the raw materials primarily determine the quantities used. Cottonseed oil consumption in margarine has decreased, and in mellorine has leveled off since 1955. Mellorine manufacture and sale are now authorized in 12 states.

Cottonseed oil is also used in candy, ice cream, and as a packaging oil. The production of food fats in 1958-59 is up sharply. This means increased competition for cottonseed oil and probably more difficulty in the disposal of the darker colored refined oils.

Cottonseed Oil "Foots"

Cottonseed oil "foot," the byproduct of the oil refining operation, is used in a variety of products--for soap, glycerine, fatty acids, and as a feed additive. Between 1945 and 1953 consumption of vegetable oil foots in soap manufacture declined percentage-wise from 32% to 18% of total consumption, creating a need for new uses of this potentially valuable product to the extent that its disposal as waste was necessary by some refineries.

Now, the largest single use of cottonseed oil foots is in fatty acid production, for which it has been estimated one hundred million pounds of cottonseed foots are consumed annually. The fatty acids are used in the manufacture of a wide range of industrial products including food emulsifiers, pharmaceutical products, insecticides, fungicides, cosmetics, rubber, plastics, and finishes for leather, paper and textiles. Another new market opened for cottonseed oil foots with the advent of solvent extraction. It is now blended into cattle feed as a fat and mineral supplement. It has been reported that the annual consumption in the Fresno cattle feed lot alone is about one-third of a million pounds.

Cottonseed Meal

We wrote a number of our friends at the National Cottonseed Products Association, in industry, and at State Experiment Stations in order to obtain accurate information on the cottonseed meal situation in localities throughout the cotton belt states.

From replies received, we can say that cottonseed meal is competitive with soybean meal in mixed feeds, and this use of cottonseed meal is increasing in California, the southwestern states, and in the extreme southeastern states, in which areas it is generally less expensive than the soybean meal. It appears that there could be a market for degossypolized cottonseed meal for most of the vegetable protein supplement in laying feeds throughout these areas.

During the past two seasons, more than 2 million tons of cottonseed meal were used in feeds. It is believed that 80 to 90 percent of this goes into manufactured feeds. This percentage will vary depending upon the relative prices of cottonseed and soybean meals from Oklahoma south. When cottonseed meal is cheap in relation to soybean meal, more of it is used as straight cottonseed meal in the form of cracked cake and pellets during the range cattle feeding season. Increasing amounts, estimated at 120,000 tons, are going into poultry and swine rations (including those for laying hens) in California and Arizona.

Other uses of cottonseed meal are in flour and for fertilizer.

New Uses of Cottonseed Oil Products

In mentioning new uses of cottonseed oil, the importance of the interconvertibility of fats and oils through chemistry, and the resulting proximity of oil prices in recent years cannot be over-emphasized. Yesterday, there was a consideration of what would benefit a particular oil, but today, there is the one of what will benefit all fats and oils.

At the Southern Regional Research Laboratory, cottonseed oil was reacted with acetic acid to produce acetoglycerides which have unique and potentially valuable properties. These properties include the ability of the solid product to remain quite plastic in the solid state. Acetoglycerides are produced commercially from fats in the United States and England, and are marketed for use in cosmetics. Their use with foods depends upon approval by the Food and Drug Administration. Food uses under consideration are: in a global edible spread for the Armed Forces, as coatings for all kinds of food products, and as plasticizers for various kinds of resins--particularly for plastic, to be formed into sheets or film and used as a wrapper for food products.

A process has been developed at SU on a continuous prepilot plant scale to convert the fatty acids present in cottonseed foots directly to their esters. The cottonseed foots are reacted with methanol and the crude product distilled to recover the esters. This development should enable the production of the esters at lower costs and consequently make them economically attractive for many uses. Such a development would provide a new outlet for the foots and thereby contribute to the stabilization of the market for this commodity.

Researchers at our Laboratory are developing a process for producing an inexpensive cocoa butter-like fat from hydrogenated cottonseed oil and triolein. The potential for such a product is best illustrated by the fact that in 1957, 13.5 million pounds of cocoa butter were imported, with a value of 7.85 million dollars. The need for this new product is further emphasized by the fact that during the past summer, cocoa butter sold for about one dollar per pound.

CLEANING OF COTTONSEED

By

E. L. D'Aquin

Southern Utilization Research and Development Division

Since the last meeting, efforts were concentrated on further development of the inclined horizontal moving belt device, which had been originally designed to clean the high-trash fractions produced by the projector unit, but which proved highly effective in cleaning straight cottonseed. The device utilizes an entirely different principle of separation from conventional methods. The first unit consisted of a moving endless belt covered with a rough textured rubber surface and inclined at an angle of about 30°. Active portion of the belt measures 3' x 5'. Operating principle is based on the trash and seed having differential properties of rolling, bouncing, and adhering. Typical results at a 3T/day capacity rate showed removal from very trashy cottonseed of about 55% of the total foreign matter, 70% of the stems, 50% of the boll walls, and 90% of the shale.

Based on the above results, a larger machine, 8' x 10', was fabricated and placed in operation. A series of preliminary runs with trashy cottonseed was then conducted as required to test operate, to standardize on feeding and product collecting procedures, and to establish practical upper and lower limits for the three principal variables (belt angle, belt speed, and capacity rate) to be investigated in a set of twenty statistically designed experiments. Results of these experiments, when completed, will be subjected to statistical analyses, and should establish optimum operating conditions for best cleaning performance.

Preliminary results with this machine to date (even though optimum operating conditions are not yet known) show that it is very efficient in removing stems (up to 92%) and shale (up to 100%) from linty, trashy cottonseed at capacities in line with those of conventional oil mill cleaners. On typical single-pass test runs on high-linters seed with 2.4% trash content at a capacity rate of about 25T/day, about 71% of the total foreign matter and 93% of the stems were removed from about 94% of the seed. At 50T/day capacity about 55% of the foreign matter and 75% of the stems were removed from 95% of the seed, in one pass, and a total of 68% of the foreign matter and 91% of the stems after two passes. On cleaned seed (after oil mill cleaners) of 1.08% trash content in one pass at a capacity of 25T/day, foreign matter removed from 90% of the seed was about 50%, and stems about 80%. When operated at about 50T/day capacity on delinted seed (after oil mill first cut) having 0.70% trash, trash removal from 96% of the seed was about 39% and stems 63% in one pass, and 51% of the trash and 75% of the stems after two passes. All of the above reductions are reported on a dust- and sand-free basis and would be higher if the sand had not been previously screened out of the seed.

A new technique which employs flaming such as is used at planting seed gins, was described for recovering practically 100% of the seed that are trapped in the trash-seed fraction that contains all of the trash that is removed. This fraction has averaged about 3-6% by weight of the feed to the belt cleaner. The procedure comprises the application of flames to the mixture as it is suspended on a screen. This burns off the excess cotton lint and agglomerates which mat together the trash and linty seed, and thus renders it free-flowing. The flame-treated mixture is then passed on the belt conveyor unit to recover the seed almost completely from the trash. This method appears simple and inexpensive, and one that can feasibly be carried out on a continuous basis at an oil mill. Substantial recovery of this seed should make possible the production of two products, one the acceptably cleaned seed, and the other the removed trash practically seed-free.

Results were presented of experiments designed to evaluate the possible advantages and feasibility of recycling a portion (14%) of the cleaned seed product which was relatively high in trash content. It was concluded that recycling of the particular fraction did not adversely affect the improved cleaning of the final cleaned seed stream, but did reduce the weight of the final trash-seed fraction to be flamed.

The 8' x 10' ARS conveyor belt unit, as described at this meeting, has proved to be capable of removing stems and shale from gin-run cottonseed to a greater extent, and at comparable capacities, than those obtained by conventional oil mill cleaner units. Equally good cleaning results were also obtained with cottonseed after it had been cleaned by a standard oil mill cleaner, and also on mill cleaned cottonseed after it had been first cut delinted.

Performance of the machine up to now is considered strictly preliminary. At this stage of the work, it is felt that cleaning ability and capacity can be improved significantly through a better understanding of the operating variables, by providing a more effective opener-feeder, and by the selection of a more suitable material, or materials, for covering the belt surface.

It is planned at an early date to make the machine available for installation in an oil mill so as to be able to evaluate it under actual mill conditions.

Trade sources believe that the production of cleaner linters insures the best monetary return to the oil mill operator, and thence to the farmer. It is felt that the development of this new machine brings our research on seed and linter cleaning closer to practical application and commercialization.

CLEANING OF COTTONSEED by J. R. Hamlett, Valley Machinery & Supply Co., scheduled at this time was not presented due to Mr. Hamlett's inability to attend the Clinic.

USE OF PURIFIED COTTON LINTERS
IN MANUFACTURE OF CELLULOSE ESTERS

by

F. M. Volberg
Tennessee Eastman Corporation

This paper will cover the use of purified cotton linters in production of cellulose esters at the Tennessee Eastman Company. It will discuss the need for high-quality linters and for control of radioactive contamination.

The Tennessee Eastman plant at Kingsport, Tennessee, is one of the largest cellulose esters plants in the world. It is by far the most diversified, with important products for yarn, plastics, photographic film, cigarette filters, and outside sales to manufacturers of a wide variety of products. Our esters include secondary acetate, triacetate, acetate-butyrate, acetate-propionate, and propionate esters. In all, there are twenty-two different types. We will discuss the use of cotton linters in the manufacture of the principal products.

Use in Plastics

To be a good plastic raw material, purified linters should have these properties:

1. The linters should produce a plastic product having low color.
2. The plastic product should have a minimum discoloration due to heat in molding, extruding, or forming. This applies to normal heating and also to excessive heating, possible under certain molding practices.

3. The linters should have uniform reactivity in acetylation and should be of uniform physical form.

The best way to test the quality of a sample of linters is to go through the steps of esterification, hydrolysis, precipitation, washing, stabilization, and drying to make the cellulose ester. This is then compounded into a plastic. Color, heat stability, and other properties of the plastic are then measured. This can be done with some success in a well-planned pilot plant. It is much better to carry out the whole test on plant scale.

Once a source and grade of linters or wood pulp is established, it is most practicable to use simple laboratory tests for quality control purposes. We measure color and haze by a bottle acetylation test (1). A more refined vacuum bottle acetylation test (2) shows overall reactivity and reactivity of the more resistant fibers. Periodic analyses are made for cuprammonium viscosity, alpha cellulose, ether extractables, ash, soda soluble, and iron content. Sheet linters are tested for porosity and Mullen strength.

The simple quality control tests are seldom considered to be true measures of product quality where linters are of different grades and from different bleacheries. For an established grade and a single source, however, the simple tests, like the bottle acetylation, are very useful in showing deviations from normal quality. These deviations will generally show up in the finished plastic.

During the last few years, there has been an increasing emphasis on the need for top quality linters for plastics use. This is just a fight for survival in an increasingly competitive field. Some years ago, polystyrene threatened growth of the cellulose ester plastics. Now there is vigorous competition from polystyrene, polyethylene, polypropylene, modified styrenes, modified acrylics, and butadiene-polymer blends. Most of these new plastics are opaque or dark colored so they compete mostly in colored and pigmented products. Manufacturers of cellulose ester plastics have had to stress clarity and low color since more of their sales have been in clear plastics.

Improved wood pulps have caused a situation where purified linters must be of highest quality to have any advantage. The wood pulp plastic is really quite close to linters in color and clarity. There is no difference in strength, toughness, and other physical properties.

Use in Photographic Film

Another important use of purified linters is in the manufacture of cellulose esters for photographic film. There are several different esters made from both linters and wood pulp. These include cellulose triacetate for motion picture and TV film, cellulose acetate butyrate for X-ray film, cellulose acetate propionate for aerial photography, and secondary acetate for certain film uses.

Cotton linters must meet the following requirements to be suitable for use in film manufacture:

1. Chemicals used in purification of the linters must be controlled to avoid photosensitive difficulties from certain trace contaminants.
2. Acetylation reactivity should be controlled and uniform. Products should have high filterability.
3. There should be a minimum of contamination with radioactive dust from atomic bomb tests.

This last requirement may easily be the most important one today because radioactive contamination is threatening to curtail use of linters for photographic film. At this point, it should be emphasized that photographic film can be damaged by very small amounts of bomb dust that do not approach the danger level for human beings. One can assume that the contamination takes place any time the lint fibers come in contact with large quantities of air. This could be during ginning, delinting of seeds, or in handling and drying at the bleacheries. Naturally, the condition of the air has a direct relationship to the amount of contamination picked up. For the last few years, there has been much more bomb dust in the air from June to January than during the other half year. Unfortunately, this covers the ginning season and most of the delinting season. The purification steps at the bleacheries undoubtedly remove some of the contamination. However, some of it goes all the way through to the finished photographic film and causes film spots. These spots can cause serious difficulties in many film uses, particularly in X-ray films for medical use or for checking industrial castings.

The first competition to contaminated linters comes from wood pulp. The raw wood is uncontaminated when it is fed to the pulp mill so control of radioactivity is simplified. However, there are still problems with wood pulp, so use of synthetic polymers must be considered.

The future of cotton linters in film manufacture depends on many things. A decrease in bomb testing would help. Our company has spent large sums of money in protecting the materials in cellulose ester manufacture and film production. The bleacheries may have to consider similar steps. A program has been started to tag the bales of raw linters to show date of delinting. This will allow selection of linters used in purification runs intended for photographic use. Selection will be based on general atmospheric conditions at time of delinting. The Eastman organization has set up twenty-five monitoring stations located at widely scattered points in the United States and western Canada. At these stations, air is being filtered twenty-four hours a day and tested for radioactive contamination. Data from these tests, plus a knowledge of air flow patterns, allow a selection

of raw linters based upon probable contamination. Recent production lots of purified linters have been rather badly contaminated because there have been no "good" delinting dates for the last several months. The choice is between "poor" and "bad." Quality of film products has been maintained by use of lint inventories and by substitution of wood pulp.

Use in Acetate Yarn

Acetate yarn once consumed most of the linters used in cellulose esters. For years there has been a trend toward substitution of wood pulp. This has been due in part to better filterability of wood pulp esters and to fluctuations in the price of purified linters. For use in yarn, linters should give high filterability, low color, and good yarn properties. Some of the desirable yarn properties are hard to measure or even to describe, so evaluation of raw materials is mostly left to the companies producing the yarn.

Use in Other Products

Another use of linters is for production of a wide variety of cellulose esters which are sold to producers of plastics, plastic sheeting, lacquers, hot-melt formulations, wire coatings, etc. Requirements here are similar to those for linters to be used in plastics.

In summarizing the use of cotton linters in manufacture of cellulose esters, we must examine each major product class. The cellulose ester plastics have shown past growth and are continuing in high volume production against strong competition from a number of old and new plastics. This has been possible because of many steps to improve quality and reduce costs. In spite of continued dollar inflation, there has been no appreciable increase in price of acetate and butyrate plastics in the last ten years. Cotton linters are the preferred raw material, but improved wood pulps offer strong competition at stable prices. The production of photographic film consumes important quantities of cotton linters. This use is currently threatened by radioactive contamination of cotton linters before they reach the bleacheries and during purification steps. A program has been started to minimize contamination, and further steps are being taken to allow continued use of cotton linters for film products. Acetate yarn continues in good production volume, with important amounts being used in cigarette filters. However, cotton linters have been largely displaced by wood pulp in yarn production. Increasing amounts of cellulose esters are sold commercially for use in plastic sheeting, lacquers, wire coatings, etc. Both wood pulp and cotton linters are used as raw materials.

In the past, cooperation between lint suppliers and users has resulted in improved grades of purified linters. Further improvements and stable prices are important to insure that present quantities of linters will be used in manufacture of cellulose esters, and to make it possible for additional amounts to be used.

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PANEL DISCUSSION - AFTERNOON SESSION - FEBRUARY 16, 1959

PANEL - Smith, Allen, Moderator
Decossas, K. M.
D'Aquin, E. L.
Volberg, F. M.

Mr. Smith, the Moderator, emphasized that these meetings are very worthwhile, but there is a need for a "letting down of the hair" and more group participation in the discussion period. He asked that half the group turn their chairs toward him and that instead of a formal panel discussion that there be a discussion by anyone who has anything to say. Of course, the speakers are available to answer questions. A well-known mill superintendent, Charlie Rankin of Texas, has used this informal approach to teach through such group discussions.

Question: How do you handle "boll bases"?

D'Aquin: We don't have the final answer yet on how to handle boll bases. Some go along with the seed. We can remove others, for example, when they are attached to sticks. We would like to know the relative importance of boll bases and boll walls to the cleaning problem.

We believe that foreign matter in the feed for first cut linters must be below 0.5% to get a high grade linters product.

We have been able to remove 50% total foreign matter from seed after the first cut with our machine. You will have an opportunity to see it in action tomorrow afternoon when we will be running west Texas "high trash" seed.

D'Aquin: How important is it to clean up the fraction which passes through the herringbone screen?

Verdery answered that one would have to see the fraction to know what's in it.

Wamble stated: If this trash is taken out in one place, we "have to" put it back some place else.

Milner: Don't put it back in the linters, since this would drive people to purchase wood pulp. Perhaps this machine will help to improve quality of linters. The drive to wood pulp is due to dirt and trash in linters. Let's try to improve linters further!

Gastrock: The trash thrown away in this case would amount to about 1/5 the value of oil normally left in hulls.

Allen Smith: Couldn't we put such trash in hulls and get price of hulls for it?

Wamble: Probably could.

D'Aquin: Surely trash is worth the value of hulls. The amount of seed rejected in reject fraction would amount to 2 lbs. seed per ton.

Allen Smith: Volberg, are you familiar with work to change the cellulose molecule to recapture the tire cord market by adding resiliency and make tire cord more enduring?

Volberg: No.

Milner: Only one tire cord manufacturer is using linters at the present time in the United States. The total tire cord market is 200,000 to 300,000 tons.

Five leading companies are now selling "Tyrex" for tire cord. They are American Enka, Courtaulds of Canada, American Viscose, DuPont, and Industrial Rayon. This product seems to be rayon--not called rayon--but called "Tyrex."

The manufacturers are very proud of this product.

Acetate yarn which wood pulp now holds is the best market left to linters. Linters still have inherent quality advantages in the acetate yarn market that wood pulp does not possess.

Allen Smith: A. T. Farrell has a new machine for cleaning which Mr. Hamlett went up to see. Has anyone seen it yet? (No response)

This machine will probably compete with the Bauer Brothers equipment.

Wamble: What about electrostatic separators for seed cleaning; are they being given consideration?

D'Aquin: We have given consideration to this method of cleaning. We want to try dropping a mixture down a 4- or 5-inch chute and determine the shift of seed and trash.

McCourtney: We have done a limited amount of work using a T. V. picture tube with 16,000 volts on surface. This tube acted as a large capacitor. The cottonseed as such was not affected, and the trash only slightly affected.

Ginaven: Our company, Bauer Brothers, worked with Quaker Oats on the application of their patents for about a year on electrostatic methods, and decided that there was no practical application to cleaning of cottonseed. The Phillips' patents seem to be most promising and have best characteristics.

D'Aquin: We will try to concentrate trash to about 10% before trying other cleaning methods.

Spadaro: The wood pulp people are engaged in an extensive research program to improve wood pulp so that it will come up to linters.

Spadaro: Mr. Volberg, do soil conditions where cotton is grown affect properties of linters?

Volberg: I don't know.

Dr. Loden: Linters from different areas of growth will show an accumulation of different minor elements.

Spadaro: What about iron?

Dr. Loden: Iron will accumulate in the lint.

STATUS OF RESEARCH ON DEVELOPMENT OF COTTONSEED MEALS FOR
POULTRY AND SWINE RATIONS

By

Vernon L. Frampton
Southern Utilization Research and Development Division

ABSTRACT

Eggs from hens fed cottonseed meals were examined by means of a photographic method for determining the relative discoloration of stored shell eggs. It was observed that the chromogen in the cottonseed meal eggs is a pH indicator, and that a part of the development of discoloration during storage is due to a change in the acidity of the yolk. The quantity of chromogen in the yolk increases with time of storage, and with length of time the hens are on cottonseed meal-containing rations. A regression analysis involving the extent of discoloration and chemical constituents of 27 meals indicated that the total gossypol and the severity of treatment of cottonseed are important factors in inducing discoloration, and that the discoloration is essentially independent of the free gossypol content of the meals.

PROGRESS REPORT ON DEVELOPMENT OF GOSSYPOL FREE COTTONSEED

By

Harold D. Loden
Paymaster Farm
Plainview, Texas

In this presentation I would like to cover four points regarding the possible development of gossypol free cottonseed. These points are:

1. Nature of the genetic character which makes possible the development of varieties with gossypol free or very low gossypol content cottonseed.
2. A review of work in progress on this problem from a plant breeding standpoint throughout the Cotton Belt.
3. Summary of the potential importance of this character to the cottonseed processing industry and indirectly to the cotton industry as a whole.
4. A look at some of the problems which may be expected in commercial utilization of this new development.

I. NATURE OF GOSSYPOL FREE COTTONSEED

I am sure you are acquainted generally with the development of the genetic character which makes possible the development of gossypol free varieties of cotton. The original glandless character was identified and isolated by Dr. Scott McMichael, U. S. Cotton Field Station, Shafter, California, who discussed this subject at the Seventh Cottonseed Processing Clinic last year. Gossypol found in cottonseed is generally considered as being in the pigment glands. Considered in its most elementary form, the development of gossypol free cottonseed is simply a matter of eliminating the pigment glands. The glandless line isolated by Dr. McMichael is essentially glandless and consequently very low or free of gossypol. Verification of the hypothesis that gossypol is associated with the pigment glands has been substantiated by several laboratories in which gossypol content has been found to be in relation to gland number.

Following the old theory that one picture is better than thousands of words, I have prepared two slides showing the difference between glandless and normal (with glands) cottonseed.

Slides:

Slide No. 1 - Two cottonseed kernels (magnified 8 diameters)

Slide No. 2 - Four cottonseed kernels (magnified 6-1/2 diameters)

II. WORK IN PROGRESS

In an attempt to determine the extent of work now being conducted on low gossypol research by state, federal, and private cotton breeders, I mailed a survey questionnaire to cotton breeders throughout the Cotton Belt. Results of this survey are given in Table I. The data in this Table shows that 13 individuals in 38 replies indicated no work in progress on this problem. It is of interest to note that among the 25 individuals reporting work in progress, 18 work in either federal or state cotton breeding programs while 7 are employed by private cotton breeding firms.

The survey was mailed to individuals in 14 states and answers indicate work is in progress in 13 states. Data in Table I indicate the extent to which work is done by private and institutional breeders by states.

One of the most interesting conclusions from this survey deals with the type of work being conducted. The survey indicates that at 17 locations the only interest is in an applied program designed toward developing low gossypol varieties. Two individuals report they are conducting only basic research while 6 report both basic and applied research efforts. These observations may be rather significant.

TABLE I

SUMMARY OF DATA OBTAINED FROM SURVEY QUESTIONNAIRE TO COTTON BREEDERS
RELATIVE TO CURRENT RESEARCH ON DEVELOPMENT OF LOW GOSSYPOL COTTONSEED

	<u>State and Federal Breeders</u>	<u>Private Breeding Firms</u>	<u>Total</u>
Questionnaires mailed	26	16	42
Replies received	24	14	38
Individuals doing no work	6	7	13
Individuals reporting work in progress	18	7	25
Type of work in progress:			
1. only applied breeding	11	6	17
2. only basic research	2	0	2
3. both basic and applied research	5	1	6
Work by states:			
1. questionnaires mailed			14
2. states in which work is in progress by both private and institutional research groups			2
3. states in which work is being done only by institutional research groups			8
4. states in which work is being done only by private research firms			3
5. states in which no work is reported in progress			1

Available experimental data indicate a lack of knowledge regarding the exact genetic nature of the glandless character. Likewise, little information has been accumulated regarding the physiological relationship of the glandless character to yield and other agronomic characters. It appears that possibly both institutional and private cotton breeders may have become too engrossed in the "romantic" aspect of developing gossypol free varieties. I would suggest that the Cotton and Cottonseed industry as a whole would profit from more basic research designed to develop fundamental information relative to all phases of the problem. The need for more basic information is implied in the answer to a question of the questionnaire requesting an estimate of time involved before low gossypol strains might be considered for commercial release. The average reply indicated 7 to 10 years would be required with some replies indicating as much as 20 years or an indefinite length of time. Such statements indicate that the best approach to reduction of this time interval may be concentration by qualified individuals on development of fundamental information regarding the genetics, physiology, chemistry, etc., of the glandless character.

III. POTENTIAL VALUE OF LOW GOSSYPOL COTTONSEED

A study of this problem requires consideration of the economic value of the low gossypol character. Many of the potential advantages are familiar to you, and I will cover most of them only briefly.

1. The first advantage generally considered in any analysis of this problem is the possible universal use of cottonseed meal in the feeding of poultry and nonruminant animals. In this respect it is possible that developing glandless varieties will not completely answer the problem since possibly all of the gossypol determined on analysis is not "classic" gossypol, but partly "gossypol-like" substances. Additional basic research is needed to determine whether or not the removal of the glands in cottonseed removes both gossypol and related gossypol-like substances.
2. The second advantage in development of low gossypol varieties would be the increase in protein quality of cottonseed meal which may be possible when seed from low gossypol strains are processed at lower temperatures. Improved methods of processing should result in higher protein solubility and an over-all improvement of protein quality of cottonseed meal.
3. Probably one of the greatest potential advantages inherent in low gossypol cottonseed would be the possibility of a reduction in preparation and processing costs. This may also become a factor in processing equipment and would be related to capital investment expenditures by oil mills.

4. The development of low gossypol varieties is not expected to result in new market outlets for cottonseed oil as would be the case in cottonseed meal. Processing of experimental lots of low gossypol seed has indicated that gossypol free strains produce very light colored oil. Gossypol loses its identity, but not its properties, when it appears in cottonseed oil. It is generally related to oil color. High colors carry a discount, however, low colors do not carry an off-setting premium. A reduction in color would not bring any immediate, direct increase in oil mill revenue. However, the refinery could expect to profit from the development of low gossypol oil due to a reduction in refining costs and could be expected to pass a portion of these profits on to the trade in the form of proportionately higher bids for oil.
5. One property observed in experimental lots of oil from low gossypol cottonseed should result in immediate benefits to oil mills. At present with the newer types of processing a large proportion of the gossypol is in the crude oil. This is a major factor in the "color reversion" problem. The unstable gossypol decomposes into highly pigmented color bodies that resist normal refining and bleaching processes to a high degree. This decomposition occurs so rapidly that a delay of a day or so in refining can result in appreciable losses in oil value; consequently, mills are under pressure to get oil shipments expedited to the refinery. Such procedures are naturally expensive. It is possible that the expected reduction of gossypol content in oil resulting from development of gossypol free varieties could entirely eliminate this problem and result in further reduction of oil mill costs.
6. There are other advantages which may or may not accrue to an oil mill processing gossypol free cottonseed. Examples would be improvement in the average quality of the acidulated soap stocks and the possible removal of the source of present objections of the refineries to degummed or water-washed cottonseed oils.

In summary the advantages of low gossypol content cottonseed appear most likely to be realized in the reduction of oil mill operating costs with improved ease in processing. The second major advantage is the possibility of opening up the large volume market for cottonseed meal as poultry feed. It can be expected that price increases to the producers of cottonseed must necessarily await full evaluation of the value of such seed in cottonseed processing and cottonseed meal outlets. Possible cottonseed price increases will result from competition for raw products by the cottonseed processing industry.

IV. PROBLEMS IN UTILIZATION OF LOW GOSSYPOL STRAINS

Possibly too much has been said ~~since~~ the development of the glandless character of the potential advantages inherent in low gossypol strains. It would be advisable to look at some of the possible problems involved in order to better analyze the apparent advantages.

The first problem, of course, is the one associated with breeding and commercial release of low gossypol strains. It is self-evident that low gossypol varieties must be equal to the best commercially available varieties adapted to any production area for all characters such as yield, fiber properties, etc. We cannot overlook the fact that cottonseed is a byproduct in the production of lint. The difference in per acre return of lint vs. cottonseed makes it apparent that a small reduction in yield could not be adequately offset by a major increase in price of cottonseed.

The next problem associated with breeding is the time interval. We have previously discussed this aspect, and it is evident that low gossypol varieties are not to be expected as a reality in the immediate future. We must also recognize the necessity of accumulating more basic information rather than hurried attempts by cotton breeders to develop commercial low gossypol or gossypol free varieties.

Probably one of the greatest problems facing the industry in utilization of low gossypol varieties, when developed, is limited to that portion of the Cotton Belt east of the New Mexico line. From this point eastward the selection of cotton varieties by a farmer is still a matter of individual judgment and varieties developed by private cotton breeding firms occupy a majority of the acreage. In any area a number of varieties may be grown, and it is immediately apparent that the seed of low gossypol varieties cannot be segregated at the gin or oil mills. In areas where one variety of cotton is grown the seed could be pure at the gin, however, few, if any, oil mills would be able to maintain the identity of such seed after it leaves the gin. It must be recognized that the maximum potential profits from low gossypol varieties can be realized only after such varieties have been developed to replace essentially all present varieties. Such will greatly increase the time interval required before oil mills may expect to receive gossypol free seed in adequate volume to permit full appraisal of the economic aspect of this character. It is evident that in one variety area, such as the San Joaquin Valley of California, where variety planted is determined by law, this problem would be minimized.

In summarizing disadvantages and problems it may be stated that a considerable length of time is to be expected before acceptable gossypol free varieties are developed. Likewise, it must be expected that a still greater time interval will be necessary before the total economic importance of this development can be evaluated by the oil mills.

SUMMARY

In this discussion I have tried to bring you up to date on the current status in development of gossypol free varieties of cotton. I think we will all admit that a critical study of this problem does not reveal the picture to be as "rosy" as many of us may have expected. Major problems must be solved both by cotton breeders and oil mills. The economic aspects are still unevaluated. Even in view of these somewhat discouraging remarks, we must consider the development of basic gossypol free breeding material to be one of the major cotton breeding advancements of this century. The potential value of this character to all segments of the cotton industry must be considered enormous. We must continue, and expand, our efforts to fully capitalize on the potential value of this discovery, and at the same time recognize that we may have to adjust our thinking regarding the time interval involved.

INFLUENCE OF PROCESSING CONDITIONS AND TREATMENTS ON COTTONSEED OIL QUALITY AND PROPERTIES

By

Frank G. Dollear
Southern Utilization Research and Development Division

ABSTRACT

Some of the factors in processing affecting oil quality are reviewed. Studies which have been reported have resulted primarily from cooperative research on processing in commercial oil mills or research carried out under contract. Oil quality depends a great deal on seed characteristics, that is, high or low gossypol, prime or high acid, or field damaged. Of the four processing methods, that is, hydraulic, screw press, prepress-solvent, and direct solvent, each differs in factors affecting oil quality. Oil quality factors include free fatty acids, content of gossypol-like pigment, refined oil color, bleached oil color, reversion characteristics, and oxidized acids.

Factors affecting the quality with each type of processing are discussed. These include preparation of cottonseed meats; time, temperature, and rate of cooking; and conditions of pressing or extraction.

In screw pressing a thin flake, 0.010 inch, is recommended for production of the best quality oil. Low temperature cooking and pressing also improved quality as did increase in throughput.

Foots should be recycled through the press only, and contamination or leakage of gear lubricant in the press should be avoided if highest oil quality is desired.

Temperature is very important with regard to oil color and excessive temperatures must be avoided, particularly in such processes as stripping solvent from miscella in prepress-solvent or direct solvent processing.

We are currently investigating the reaction of gossypol in cottonseed oil which causes it to change and become more difficult to remove during refining and bleaching. An improved method of oil color measurement is also under investigation.

PANEL DISCUSSION - MORNING SESSION - FEBRUARY 17, 1959

PANEL - Norris, Frank, Moderator
Frampton, V. L.
Loden, Harold
Dollear, F. G.

Norris: We will open the discussion with Dr. Frampton's paper entitled "The Status of Research on Development of Cottonseed for Laying Hens and Swine." On this subject of yolk discoloration, is the color the only objectionable factor or does the taste also become objectionable?

Frampton: The appearance of these eggs are such that I feel sure no one would venture to taste them.

Norris: Are we talking about the discoloration of the yolk after long storage or discoloration of eggs sold on a commercial basis within about thirty days?

Frampton: Color begins to appear after about three months and gets progressively worse. There are two factors involved, the chromogen and the pH factor. The longer an egg is stored the more alkaline the yolk becomes due to exchange through the vitelline membrane from alkaline white to acid yolk tending toward an equilibrium. The more alkaline the yolk becomes, the more color develops.

Norris: Did I understand you to say that as the yolk becomes alkaline a color develops? Could pH be a factor in the discoloration?

Frampton: The causes for the increase in alkalinity are the loss of CO₂ and the distribution of electrolytes which result in a change in the pH. The migration of the anions which could produce a change in the balance, could produce color. If the pH balance of the egg is not changed, the egg would not change color.

Castrock: This is just an observation but I've noticed that when an egg is hard-boiled the yolk gets a thin outer greenish layer on it.

Allen Smith: Can a dye be included in a good meal that could be used to neutralize the coloring effect of the meal?

Frampton: It may be possible that some sort of dye could be used by increasing the absorption in the red area masking the absorption in the blue green area, therefore, no color change would be noticed.

Rose: Color film is panchromatic and will vary from batch to batch, what effect would this have on the result obtained on analysis of the film spectrophotometrically?

Frampton: The film colors vary and may cause some color distortion, but we use it to eliminate the human factor since it's the best approximation we have to date. We try to minimize film variation by ordering film from some batch and have it processed at same time and under identical conditions of developing.

D'Aquin: Does color develop in eggs from hens fed meal after it has been stored six months? Do eggs stored in either frozen or powdered form discolor after storage?

Frampton: We have no data available that would answer either question.

Spadaro: Does eating eggs known to develop color in storage have any detrimental effect on the human body due to the possible cumulative effect of the gossypol?

Frampton: Present knowledge seems to indicate that humans are much more tolerant to gossypol than lower animals. This is shown by nutritional work in South America where children have been fed cottonseed meal with no ill effects. It is also shown in the reports of Bernal Diaz who as a member of Cortez Conquistadores stated that Aztec priests ate raw cottonseed and nothing else.

Norris: If there are no more questions we will now discuss Dr. Loden's paper "Progress Report on Development of Gossypol Free Cottonseed."

Norris: I recall that in your talk you stated that with development and use of this gossypol free cottonseed that mills would be able to save money. Where or in what area would these savings be?

Loden: That statement is based on information from the processing industry. Savings would come from use of lower temperature in processing because the need for high temperature high moisture cooking for gossypol binding would not be necessary.

Norris: I understand that oils made from these gossypol free seeds do not revert on storage?

Frampton: That is correct.

Piccolo: I understand from Dr. McMichael that enough seed is being produced for farmers to plant on a commercial basis.

Loden: That is true. The quantity now produced is sufficient for that purpose.

Dollear: Is the fiber from the glandless cottonseed acceptable?

Loden: The fiber is completely acceptable.

Smith: Is there a relation between the amount of pigment glands in kernel and the yield of cotton per acre?

Loden: There is no evidence of decreased cotton yield due to absence of glands in the kernel.

Dollear: If you could get the production of this seed up, would quality be good or as good as the best commercial varieties?

Loden: Yes, but 15 to 25% below the best commercial varieties in production of lint or fibers.

Sale: Are soil, air, or other climatic factors involved in seed quality?

Loden: Mr. Hopper's group worked on that problem.

Pons: The gossypol content of seed of Upland cotton, for example, is a varietal characteristic. Gossypol content almost equally influenced by conditions of environment and variety. When any variety of cottonseed is planted in various sections of the country it will show varying amounts of gossypol content. The variation will depend on environmental conditions which include soil and climate.

Loden: Glandlessness is also a varietal characteristic. However, glandless cottonseed would still be glandless regardless of the location of planting because the characteristic gland producing factor is absent.

Piccolo: On analyzing glandless cottonseed meal, I've been getting 0.01% free gossypol, even though no glands are visible in the seed.

Loden: Your analytical results are probably correct. The absence of glands is a recessive characteristic. Cross pollination by bees may account for the development of some gland containing seed.

Piccolo: The seed to which I refer was protected seed, hand pollinated.

Frampton: Color is sometimes a fictitious thing, small and not measurable. What is measured could be background color due to reagents used.

Dollear: Pollination by bees would not affect the gland production in the kernel in the same generation would it?

Loden: Yes, it would since glands would be visible in the cotyledons of the plant on growing (F1 generation), therefore, glands would be present in seeds being crushed.

Piccolo: What is the source of Dr. Rhine's North Carolina glandless seed?

Loden: That also has some Hopi variety in it but not much.

Norris: If there are no more questions we will next discuss Mr. Dollear's paper "Influence of Processing Conditions and Treatment on Cottonseed Oil Quality and Properties."

Wamble: The work reported under contract with U. S. D. A. was completed at about the time work was started with hi-speed screw presses. Although all this work is interesting it is not applicable to hi-speed screw presses. Consequently some work is needed to develop information for use with hi-speed presses.

Pons: A lot can be done to produce a better oil; however, none of these things permit the production of better meals. This conflict of purpose makes it difficult for an oil mill to decide what they should do.

Norris: If one multiplies oil yield by cents per pound and the same with meal, processor can see which is more important. Treatment with additives to remove gossypol gives oil of bad color. It's a pretty complicated situation.

Dollear: People look to glandless seed as a hope, both for good oil and good meal.

Verdery: Whatever is done in processing to improve meal, such as, cooking meal to flush oil from it, has an effect. Wamble has a process for a good meal but gives a bad oil. We could do something to oil to improve it such as quick treatment or partial refining or miscella refining.

Frampton: Not much is being done especially when dealing with poor seed, therefore, the color becomes fixed. If gossypol was removed quickly a good oil could be made.

Wamble: A low gossypol meal is produced by treatment of meats with amines such as "octylamine" using the ratio of four moles of amine to one mole gossypol. Slurry in oil or water and impregnate the meats, then roll into flakes, and then cook mildly. Either solvent extract or screw press can be used on oil extraction. Another method is to treat prepared meats with amine. The gossypol amine compounds go into the oil where the real problem is to try to remove by miscella refining the oil. The oil could be treated with H_2SO_4 , but this is too drastic a treatment for an edible product.

The meal has protein solubility of 70-90%, free gossypol of 0.03-0.08%, and total gossypol of 0.1-0.3%. The meals were tested and found to be nontoxic to chicks.

Allen Smith: Mr. Wamble, why couldn't you make two passes through the press - one to make good oil, then treat meal and repress.

Wamble: It is possible. Such cottonseed meal has a similar appearance to soybean meal.

Frampton: Not very much information is available in the literature on toxicity of amines. Indications are that most certainly aromatic amines are very toxic but only some of the aliphatic amines may be toxic. The problem should be settled by more research. Binding is on the amino acid group substituting gossypol in the linkage so some of the amine is left in meal.

Gossypol has properties of phenols and with oxidation it forms quinones causing red color. Linking with amine prevents this oxidation product as well as the change in color. The amines capable of doing this are amines having structure such as diethylene diamine.

Wamble: Search literature on toxicity of amines, little available. Process using these materials must be cleared with pure food and drug laws.

Frampton: Another problem is reaction of amine with fat forming a glyceride amide. In this way amine is fixed in oil and is very difficult to get rid of.

RESOLUTIONS

1. Be it Resolved - That we express our appreciation to Lawrence Hodges and his program committee composed of Messrs. Brawner, Fowler, Castrock, Long, Norris, Smith, Quinn, and Verdery, for a most interesting and instructive program; also to Ernest J. Rice, Jr., President, Clarence Garner, Secretary, and R. F. Patterson, Chairman of the Research Committee of the Valley Oilseed Processors' Association and all who have appeared on our program.

2. Be it Resolved - That we extend to Dr. C. H. Fisher and his staff our appreciation for their continued interest and their valuable contributions to this Clinic, and for making us feel so welcome.
3. Be it Resolved - That we recommend the appointment of a Committee for Long Range Planning - such committee to outline a long range approach looking to the ultimate solution of the problem relating to unexplained oil losses appearing at unpredictable times. It is further recommended that J. H. Brawner be appointed Chairman of this Committee.
4. Be it Resolved - That the promising results obtained in the study of the problem of cleaning cottonseed warrant a continuation of this project toward a commercial application of the ultimate findings.
5. Be it Resolved - That, as a result of these Processing Clinics, the industry has been able to increase its efficiency, contributing to increased prices of cottonseed to farmers, and improved products to the consumers. In our opinion these Clinics should be continued each year.

Resolutions Committee:

T. H. Baker, Jr., Chairman

J. R. Mays, Jr.

Zach McClendon

February 17, 1959

UNITED STATES DEPARTMENT OF AGRICULTURE
AGRICULTURAL RESEARCH SERVICE
SOUTHERN UTILIZATION RESEARCH AND DEVELOPMENT DIVISION

Eighth Cottonseed Processing Clinic
at the
Southern Regional Research Laboratory
New Orleans, Louisiana

in cooperation with

VALLEY OILSEED PROCESSORS' ASSOCIATION, INCORPORATED

P R O G R A M

February 16, 1959 - 9:30 a.m.
Auditorium - Third Floor

Ralph Woodruff, VOPA, Chairman

I. 9:30 a.m. Opening Remarks

G. E. Goheen, Asst. Director, SU
E. J. Rice, Jr., President, VOPA

II. 10:00 a.m. Sampling of Product
and Product Control

M. H. Fowler
The Buckeye Cotton Oil Co.

10:30 a.m. Intermission

III. 10:50 a.m. Determination of Residual
Solvent in Meal

J. J. Spadaro
Engineering and
Development Laboratory, SU

IV. 11:10 a.m. Mill Scale Material
Balance

J. H. Brawner
Wesson Oil and Snowdrift

V. 11:30 a.m. Panel Discussion

M. C. Verdery, Moderator
Anderson, Clayton & Co.

12:30 p.m. Lunch at SURDD

February 16, 1959 - 1:30 p.m.

E. L. Patton, SU, Chairman

VI. 1:30 p.m. Report on Current Utili-
zation of Cottonseed Oil
and Meal

Kenneth Decossas
Engineering and
Development Laboratory, SU

VII. 1:50 p.m. Cleaning of Cottonseed

- a. E. L. D'Aquin
Engineering and Development
Laboratory, SU
- b. J. R. Hamlett
Valley Machinery & Supply Co.

VIII. 2:30 p.m. Current Status of Linters
Utilization

F. M. Volberg
Tennessee Eastman Co.

IX. 2:50 p.m. Panel Discussion

Allen Smith, Moderator
Perkins Oil Company

February 17, 1959 - 9:30 a.m.

Robert F. Patterson, Chairman
Research Committee, VOPA

Lawrence H. Hodges, Chairman
Program Committee, VOPA

X. 9:30 a.m. Status of Research on
Development of Cottonseed
Meals for Laying Hens and
Swine

V. L. Frampton
Industrial Crops
Laboratory, SU

XI. 9:50 a.m. Progress Report on
Development of Gossypol
Free Cottonseed

Harold Loden
Anderson, Clayton & Co.

10:10 a.m. Intermission

XII. 10:30 a.m. Influence of Processing
Conditions and Treatment
on Cottonseed Oil Quality
and Properties

F. G. Dollear
Industrial Crops
Laboratory, SU

XIII. 10:50 a.m. Panel Discussion

Frank Norris, Moderator
Swift & Co.

XIV. 12:00 N Report of Resolutions Committee

XV. 12:15 p.m. Resume' and Announcements

ADJOURNMENT

12:30 p.m. Lunch

1:30 p.m. Pilot Plant Demonstra-
tion of New Cottonseed
Cleaning Developments

E. A. Gastrock
Engineering and
Development Laboratory, SU

Visits with SURDD Personnel

UNITED STATES DEPARTMENT OF AGRICULTURE
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February 16-17, 1959

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VALLEY OILSEED PROCESSORS' ASSOCIATION, INCORPORATED

ATTENDANCE LIST

Tom E. Allen, Southern Cotton Oil Co., New Orleans, Louisiana
R. F. Anderson, Delta Cotton Oil Company, Jackson, Mississippi
T. H. Baker, Jr., Trenton Cotton Oil Company, Trenton, Tennessee
W. D. Baldwin, Hercules Powder Company, Memphis, Tennessee
J. P. Barnett, Jr., Opelousas Oil Mill, Opelousas, Louisiana
R. C. Barton, Forrest City Cotton Oil Mill, Forrest City, Arkansas
J. H. Brawner, Wesson Oil & Snowdrift Company, New Orleans, Louisiana
D. K. Bredeson, French Oil Mill Machinery Company, Memphis, Tennessee
A. H. Burner, The French Oil Mill Machinery Company, Piqua, Ohio
C. H. Caldwell, West Memphis Cotton Oil Mill, West Memphis, Arkansas
C. R. Campbell, Charles R. Campbell Company, Dallas, Texas
W. Campbell, Mississippi Oil Mill, Inc., Hollandale, Mississippi
G. E. Covington, Magnolia Cotton Oil Company, Magnolia, Mississippi
R. T. Doughtie, Jr., USDA, AMS, Memphis, Tennessee
W. A. Durham, Sr., Tristate Blow Pipe Company, New Orleans, Louisiana
L. H. Fleming, Jr., DeSoto Oil Company, Memphis, Tennessee
M. H. Fowler, Procter & Gamble, Ivorydale, Cincinnati, Ohio
A. W. French, The French Oil Mill Machinery Company, Piqua, Ohio

H. Fryer, Bauer Brother Company, Springfield, Ohio
D. E. Gandy, National Cottonseed Products Association, Ruston, Louisiana
Arno Goetz, Reis & Company, Inc., Dallas, Texas
C. E. Garner, Valley Oilseed Processors' Association, Memphis, Tennessee
A. Geismar, Geismar & Company, Inc., New Orleans, Louisiana
M. E. Ginaven, The Bauer Brothers Company, Springfield, Ohio
W. Godchaux, Jr., National Blow Pipe & Mfg., Company Inc., New Orleans, La.
H. O. Graebe, Carver Cotton Gin Company, East Bridgewater, Massachusetts
J. H. Haas, Jr., Tristate Blow Pipe Incorporated, New Orleans, Louisiana
G. A. Harper, National Cottonseed Products Association, Dallas, Texas
W. H. Harrison, Tennessee Eastman Corporation, Kingsport, Tennessee
C. W. Hasen, Chickasaw Oil Mill, Memphis, Tennessee
C. Hay, Anderson, Clayton & Company, Inc., Houston, Texas
L. H. Hodges, Barrow Agee Laboratories, Inc., Memphis, Tennessee
N. Howard, Yazoo Valley Oil Mill, Greenwood, Mississippi
A. Jenkins, Delta Cotton Oil & Fertilizing Company, Jackson, Mississippi
E. C. Kontz, Davidson-Kennedy Company, Atlanta, Georgia
W. P. Lanier, Buckeye Cellulose Corporation, Memphis, Tennessee
Rene J. Lazare, Jr., Southern Cotton Oil Company, New Orleans, Louisiana
H. D. Loden, Paymaster Farms, ACCO, Plainview, Texas
R. D. Long, Carver Cotton Gin Company, Memphis, Tennessee
J. C. Lundmark, The V. D. Anderson Company, Birmingham, Alabama
E. S. Lyle, Dyersburg Oil Mill Company, Dyersburg, Tennessee
W. C. Manley, Jr., 811 Falls Building, Memphis, Tennessee
J. R. Mays, Jr., Barrow Agee Laboratories, Inc., Memphis, Tennessee
Z. McClendon, Drew Cottonseed Oil Mill, Monticello, Arkansas

C. M. McClure, Anderson, Clayton & Company, Houston, Texas
O. M. McClure, Southern Chemical Cotton Company, Chattanooga, Tennessee
W. D. McKinney, Buckeye Cellulose, Memphis, Tennessee
N. H. Moore, 3373 Poplar Avenue, Memphis, Tennessee
E. Morris, Hercules Powder Company, Wilmington, Delaware
F. A. Norris, Swift & Company, Chicago, Illinois
R. F. Patterson, Trenton Cotton Oil Company, Inc., Trenton, Tennessee
J. D. Peier, USDA, AMS, Washinton, D. C.
C. L. Printup, Carver Cotton Gin Company, Memphis, Tennessee
T. S. Pryor, Continental Gin Company, Birmingham, Alabama
W. F. Quinn, Minter City Oil Mill, Minter City, Mississippi
A. Rose, Texas Engineering Experiment Station, College Station, Texas
O. H. Sale, Fertilizer Equipment Sales Corporation, Atlanta, Georgia
G. R. Simpson, Mississippi Oil Mills, Inc., Greenwood, Mississippi
A. Smith, Perkins Oil Company, Memphis, Tennessee
R. E. Smith, Yazoo Valley Oil Mill, Inc., Greenwood, Mississippi
W. Smith, Wesson Oil & Snowdrift Company, Inc., New Orleans, Louisiana
E. H. Tenent, Jr., Woodson-Tenent Laboratories, Memphis, Tennessee
J. L. Tennent, Delta Products Company, Wilson, Arkansas
M. C. Verdery, Anderson, Clayton & Company, Inc., Houston 1, Texas
M. Volberg, Tennessee Eastman Corporation, Kingsport, Tennessee
C. W. Wallace, The Union Oil Mill, Inc., West Monroe, Louisiana
A. C. Wamble, Cottonseed Products Research Laboratory, Texas Engineering
Experiment Station, College Station, Texas
L. J. Weber, Skelly Oil Company, Kansas City, Missouri
M. E. Whitten, USDA, AMS, Marketing Research Division, Washington, D. C.

- G. Wildrick, Lake County Oil Mill, Tiptonville, Tennessee
- A. L. Wiley, Perkins Oil Mill, Memphis, Tennessee
- E. P. Withers, Davidson-Kennedy Company, Atlanta, Georgia
- R. Woodruff, Delta Products Company, Wilson, Arkansas
- H. M. Woodyard, Nashville Cotton Oil Mill Corporation, Nashville,
Tennessee

